#11 C.C.A. 9-20-02

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of	
Charles E. FARLEY *et al.*	Examiner: Fortuna, J.
Serial No. 09/694,789	Group Art Unit: 3751
Filed: October 24, 2000	Atty. Dkt. No.: 005242.87031
For: EMULSIFICATION OF ALKENYL SUCCINIC ANHYDRIDE SIZE	

DECLARATION UNDER 37 C.F.R. § 1.131

Assistant Commissioner for Patents
Washington, DC 20231

Sir:

We, CHARLES E. FARLEY, GEORGE ANDERSON, and KARLA D. FAVORS citizens of the United States of America, do hereby state:

1. THAT we are co-inventors of claims 1-20 of the above-captioned patent application.

2. THAT prior to September 26, 2000, we reduced our invention as described and claimed in the above-captioned patent application to practice in this country as evidenced by the following:

   a) Prior to September 26, 2000 and having earlier conceived of using a starch grafted cationic acrylamide co-polymer for the emulsification of alkenyl succinic anhydride size, we directed laboratory experiments, while being employed by Georgia-Pacific Resins, Inc., to be performed, resulting in the synthesis of alkenyl succinic anhydride emulsifiers. The details of

these experiments are evidenced by the laboratory notebook pages in Exhibit A.

b) As evidenced by Exhibit A:

i. A solution of a starch grafted cationic acrylamide co-polymer having about 15 wt% solids, at about a 1.1:1 weight ratio of starch to acrylamide, was prepared. The starch used was Penford Gum 280, a hydroxyethylated cornstarch commercially available from Penford Products.

ii. The starch grafted cationic acrylamide co-polymer was prepared by charging into a reaction vessel at room temperature: 2716 g of deionized water, 494 g of acrylamide (52 wt% aqueous solution), 92.2 g of diallyldimethyl ammonium chloride (63 wt% aqueous solution, available from Ciba Specialties), and 290 g of hydroxyethylated corn starch. The pH of the reaction mass was adjusted to about 4.0 using dilute sulfuric acid. The reactants were de-aerated by sparging with nitrogen for 30 minutes.

iii. A free radical catalyst solution was prepared by combining 6.04 g of potassium persulfate and 194 g of water to form an approximately 3 wt% aqueous solution of potassium persulfate. The catalyst solution was divided into four equal portions of about 50 g each.

iv. Following de-aeration, the reaction mass was heated to 75°C. One 50 g portion of the potassium persulfate solution was added and the resulting reaction exotherm increased the reaction mass temperature to about 96°C. The second, third, and fourth 50 g portions of potassium persulfate solution were added at 8, 38, and 68 minutes after the first portion was added. The reaction temperature was maintained at about

90°C for the duration of these additions. After the fourth portion of potassium persulfate was added, the reaction mass was held for one hour, after which the reaction vessel heating was turned off. Following addition of the final catalyst charge, the reaction mass was held for one hour at 90°C, and thereafter was post-treated with a cross-linking agent.

v. A second synthesis of starch grafted cationic co-polymer was carried out in a similar manner, except that the amount of diallyldimethyl ammonium chloride was doubled to result in a doubling of the cationic charge of the emulsifier.

c) Under our direction, samples of the starch grafted cationic acrylamide co-polymer prepared as described in Exhibit A were tested for sizing ability and stability in alkenyl succinic anhydride emulsion blends comprising a surfactant. Based on the test results, the samples prepared worked for their intended purpose. The details of this testing are evidenced by Tables 1, 2, and 3 in Exhibit B and discussed below.

d) As evidenced in Exhibit B:

i. Samples of the starch grafted cationic acrylamide co-polymers, prepared according to the procedures set forth in Exhibit A (referred to as Ambond 1520 and Ambond 1530), were used to emulsify 10 wt% alkenyl succinic anhydride size solutions.

ii. In a blender, solutions of both starch grafted cationic acrylamide, as synthesized according to Exhibit A, were stirred at medium speed. A third solution of a different emulsifier (EML-2) was prepared for comparative purposes.

iii. To these three solutions, alkenyl succinic anhydride containing sodium dioctyl sulfosuccinate surfactant was added and the resulting mixture was blended at high speed for four minutes. The resulting 10 wt% alkenyl succinic anhydride emulsions using the emulsifiers in Exhibit A were fluid and milk-white, compared to the emulsion using EML-2, which was viscous and foamy.

iv. Samples of each of the three emulsions were diluted to 1.5 wt% alkenyl succinic anhydride and particle size was determined both immediately and after standing overnight. Results in Table 1 of Exhibit B showed that the emulsions of Exhibit A afforded superior performance, namely a smaller average particle size and greater emulsion stability, compared to EML-2.

v. These superior performance results were confirmed in additional emulsification experiments where different cellulosic materials were sized and differing ratios of the starch grafted cationic acrylamide co-polymer to alkenyl succinic anhydride were used. The results of these additional experiments are summarized in Tables 2 and 3 of Exhibit B.

3. THAT the emulsifier and emulsification process described above meet the limitations of claims 1-8 and 11-20 in the above-captioned patent application.

4. THAT each of the dates deleted from Exhibits A and B is prior to September 26, 2000.

5. THAT the acts referred to in Exhibits A and B were performed in the United States.

6. I declare further that all statements made herein are of my own knowledge are true and that all statements made on information and belief are believed to be true;

and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. §1001 and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: 4 SEPT. 2002

Charles E. Farley

Dated: Sept. 4, 2002

George Anderson

Dated: 8/30/02

Karla D. Favors

TITLE Hmbond 1520

Project No.
Book No. 951 D

From Page No.

Purpose: Make Cationic Amband 1520 for CEFarley for an ASA emulsifier.

	g	
H_2O #1	2080	DI
Per 290	290	96-FBB-800
Aam, 52%	494	
DADMAC	92.2	
H_2O #2	636	DI

Adjust pH from 5.78 to 4.06 w/ dilute H_2SO_4

$K_2S_2O_8$	6.04	200 ÷ 4 = 50
Water	194	

1:30 Begin N_2 Sparge
2:00 Begin heating
~~2:50~~

Time		Temp	
2:50	T0	75°C	Add 50g $K_2S_2O_8$
2:58	T08	96°C	Thick, foamy & add 50g $K_2S_2O_8$
3:28	T38	90°C	Add 50g $K_2S_2O_8$
4:28	T68	90°C	Add 50g $K_2S_2O_8$
5:18	T128	91°C	Heat off

Add KX-28 8.4g

Color - light Grey
pH 6.37
% NV 14.79, 15.35 (15.07%)
Brook Vise 444 cps

EXHIBIT A

To Page No.

Witnessed & Understood by me.	Date	Invented by [redacted]	Date
Laura Culbreath		Recorded by KDBlack	

Ambond 1520 (515D99 prepolymer)				
	PBW	Scale-up		Wt Percent
Penford Gum 290	0.0764	✓ 290.3365		7.640434753
Acrylamide (52%)	0.1297	✓ 492.9186		12.97154196
Water #1	0.5474	✓ 2079.9671		54.7359763
Sulfuric Acid (35%)	0.0002	✓ 0.9336		0.024567314
DADMAC (63%)	0.0243	✓ 92.4222		2.432164118
Water #2	0.1673	✓ 635.7530		16.73034105
	0.0000	0.0000		0
Slimetrol RX-28 (21%)	0.0022	8.4020		0.221105829
	0.0000	0.0000		0
Potassium Persulfate	0.0016	✓ 6.0205		0.15843461
Water (Pot. Persulfate)	0.0509	193.2465		5.085434064
				0
Prepolymer	1.0000	3800.0000		100
		3800.0000		0

(1530)

TITLE Armbond 1520 X2DA/DXPAE

Project No.

Book No. 951D

From Page No.

Water #1	2079
Pen 290	290
Aam	493
DADMAC	184.8
Water #2	636
Dilute H_2SO_4	67g
$K_2S_2O_8$	6
Water	193
RK-28	7.2g

pH 5.73 Adjust to 3.89 pH w 67g dilute H_2SO_4 (accidentally added 25% NaOH 1st (pH 10.3)

1:57

Time	Temp		Comment
1:57	Room		Begin N_2 Sparge
2:27	"		Begin heating
3:11	To	95°C	Add $K_2S_2O_8$ 17° exotherm in 7 minutes
3:43	T30	91°C	" "
4:11	T60	88°C	" "
5:11	T120	91°C	" "
6:11	—		Heat off

pH	6.55
%NV	16.28, 16.59
Visc	323 cps

(16442)

To Page No.

Witnessed & Understood by me. Date Inventor Date

Dana Culbreath

Recorded by KDBlack

Ambond 1520 (515D99 prepolymer)			
	PBW	Scale-up	
Penford Gum 290	0.0764	290.3365	
Acrylamide (52%)	0.1297	492.9186	
Water #1	0.5474	2079.9671	
Sulfuric Acid (35%)	0.0002	0.9336	
DADMAC (63%)	0.0243	~~92.4222~~	184.8
Water #2	0.1673	635.7530	
	0.0000	0.0000	
Slimetrol RX-28 (21%)	0.0022	8.4020	
	0.0000	0.0000	
Potassium Persulfate	0.0016	6.0205	
Water (Pot. Persulfate)	0.0509	193.2465	
Prepolymer	1.0000	3800.0000	
		3800.0000	

TABLE 1
Sizing with Novasize ASA emulsified in EML-2, Ambond 1520, Ambond 1530
Evaluation in Old New sprint

Polymer	Ratio/ASA (1)	ASA; lb / ton	HST	Particle Size, microns	Stability, 24 hours
EML-2	1/1	3.5	403	2.16	white precipitate
"	"	4	556		
"	"	4.5	794		
Ambond 1520	1/1	3.5	404	0.98	stable
"	"	4	487		
"	"	4.5	650		
Ambond 1530	1/1	3.5	464	1.37	stable
"	"	4	536		
"	"	4.5	730		

(1). The polymer/ASA ratios are those for a 20% solids Ambond.

TABLE 2

Ambond 1520 and 1530 - Sizing and Emulsion Stability

Old Newsprint Furnish - Second Test Round

Polymer	Ratio poly./ASA (1)	lb ASA/ton	HST	Particle size, micron	
				immed.	24 hr
EML-2	1/1	3.1	50	2.45	wh. Ppt.
"	"	3.4	71		
"	"	3.7	146		
Ambond 1520	1/1	3.1	66	0.93	2.99
"	"	3.4	101		
"	"	3.7	146		
Ambond 1520	0.65/1	3.1	62	1.37	1.84
"	"	3.4	133		
"	"	3.7	162		
Ambond 1530	1/1	3.1	104	1.33	4.49
"	"	3.4	-		
"	"	3.7	212		
Ambond 1530	0.65/1	3.1	181	1.02	2.89
"	"	3.4	223		
"	"	3.7	324		

(1). Ratios with Ambond are expressed as liquid resin corrected to a 20% solids product.

TABLE 3
Ambond 1520, 1530 - Sizing and Emulsion Stability Evaluation in OCC

Polymer	Ratio polymer/ASA	ASA, lb/ton	HST	Particle size, microns immed.	24 hrs
Novasize EML-2 (1)	1/1	1	27	2.31	16.3
"	"	1.4	180		
"	"	1.8	274		
"	"	2.2	983 (2)		
Ambond 1520	1/1	1	6	0.89	3.38
"	"	1.4	48		
"	"	1.8	175		
"	"	2.2	250		
Ambond 1520	0.65/1	1	7	1.05	2.67
"	"	1.4	39		
"	"	1.8	207		
"	"	2.2	434		
Ambond 1530	1/1	1	13	1.31	3.87
"	"	1.4	33		
"	"	1.8	227		
"	"	2.2	418		
Ambond 1530	0.65/1	1	35	1.33	3.17
"	"	1.4	135		
"	"	1.8	246		
"	"	2.2	494		

(1). Polymer/ASA ratio for EML-2 is liquid, as is basis (for Eml-2 at 30% solids. ratio is 0.3/1. active solids basis). For Ambond 1520 and 1530 ratio is liquid, as is basis corrected to a 20% solids resin. Thus a 1/1 ratio is 0.2/1, active solids basis. 0.65/1 is 0.13/1, active solids basis.

(2). Utterly suspect value; would expect this test value to be 400-500. Suggest ignoring.